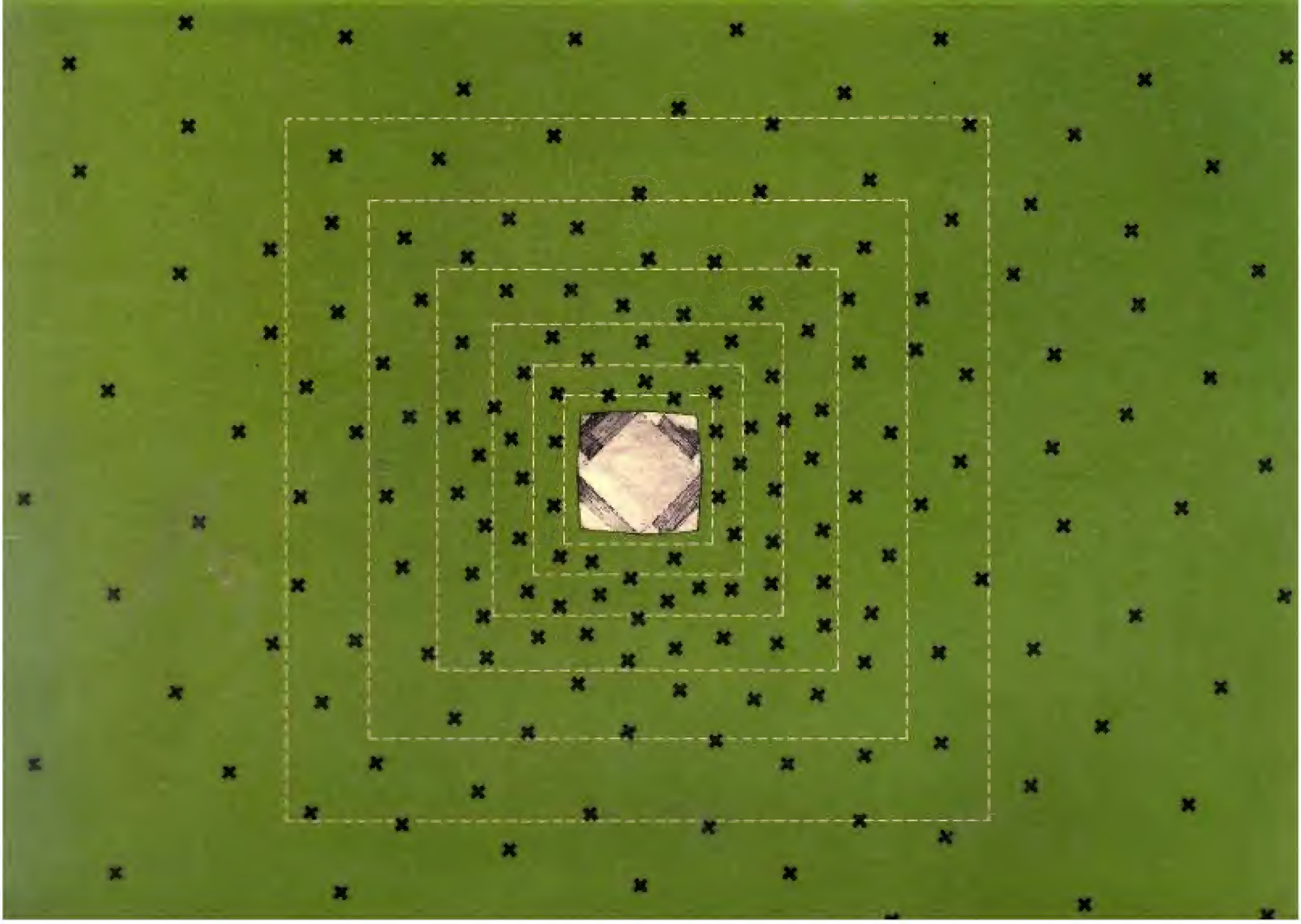
Chemistry Background Book

Growing Crystals



Introduction

You may have made some crystals of saltpetre or of alum by crystallizing a hot solution. The size of your crystals was probably quite small – from one to two millimetres across; if you are curious to know how to get bigger ones, one or two *centimetres* across or even more, this small book will tell you how it can be done. It not only gives you several recipes but also explains why they work. The last part of the book is about crystals that have grown in the rocks of the Earth's crust and describes briefly how scientists have been able to grow crystals of some of the minerals.

Growing a large crystal of potassium alum from a small seed crystal. The seed crystal was suspended on a cotton thread in a saturated solution of the alum.

Part one

Growing crystals of alum

One of the best substances for growing crystals is alum. The plan is to hang a small 'seed' crystal on the end of a thread in a solution of alum so that more alum crystallizes round it and it grows into a large crystal. To make this experiment work you must first make the right kind of seed crystal and the right kind of alum solution. The next few paragraphs give you a general idea of how it is done. Detailed instructions for doing the experiment follow in Part two, 'Growing crystals in the school laboratory', and Part three, 'Growing crystals at home'.

A saturated solution – First you must dissolve enough alum in water to make a saturated solution; that is, a solution in which no more alum will ordinarily dissolve. A saturated solution is needed for two reasons: first you will use a little of it to make good seed crystals; and then you will use the rest to make the special solution in which your seed crystal will grow.

You *could* make a saturated solution by shaking some powdered alum with the water until no more would dissolve, but it would take a very long time. A better method makes use of the fact that most substances are more soluble at higher temperatures than at lower; so if you warm the water you will get a larger quantity of alum to dissolve in it than would dissolve in cold water. When the water cools back to room temperature, you will see some of the dissolved alum reappear as crystals at the bottom. The rest of the alum will remain in the solution and the solution will then be a saturated solution.

We should mention straight away that this method can also produce a solution containing *more* alum than is contained in a saturated solution! This unusual kind of solution, a 'supersaturated' solution, occurs when the excess alum stays dissolved after the solution has cooled; no crystals appear in the clear liquid. It can only happen when the solution is completely free of foreign bodies; one speck of dust or one tiny crystal of alum will 'seed' it, causing the excess alum to crystallize out, leaving a saturated solution.

You will make a supersaturated solution later as the special solution to hang your seed crystal in, but at this stage you do not want one, so it is wise to add a little powdered alum to the solution after cooling. The tiny crystals act as seeds, and help crystallization to take place faster.

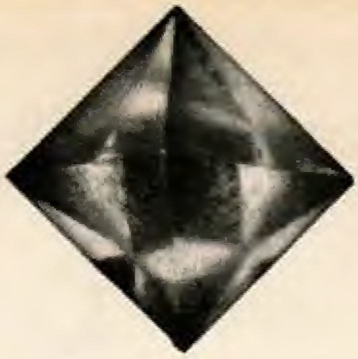
Obtaining seed crystals – To get the seed crystals you are going to use for growing, you leave a little of your saturated solution in a small basin exposed to the air, keeping the remainder carefully covered up. In a day or so crystals of suitable size and shape will appear in the basin. These are the seeds ready for planting.

Growing your seed crystals – This is done by suspending one or more seed crystals in a supersaturated solution of alum. You take the remainder of your saturated solution, supersaturate it, and hang your seed crystal in it on the end of a piece of cotton, making sure that no unwanted seeds get in the solution. The excess alum in the supersaturated solution will crystallize on your seed crystal: in other words your seed crystal will 'grow'.

Now read Part two if you are going to grow crystals in the laboratory, or Part three if you are going to grow them at home.

Part two

Growing crystals in the school laboratory



You will need the following apparatus:

1 Two 600 cm^3 beakers with no lip
2 Tin of water big enough to hold the beaker
3 Thermometer (0–100°C)
4 Reel of cotton, two sheets of thin cardboard big enough to cover the mouth of a beaker, and some Durofix
5 Bunsen burner, gauze, and stand
6 Balance
7 Measuring cylinder
8 Filter papers
9 Tweezers

To prepare the saturated solution – Clean two beakers very thoroughly. Measure out 400 cm^3 of water into one beaker and add 80 grams of powdered alum. Warm the mixture to 50°C by placing the beaker in some water in the can and heating the can over a Bunsen burner. Stir the solution well for two or three minutes. When the alum has all dissolved, pour the solution while it is still warm into the other beaker, filtering it to remove any impurities. Put it aside to cool, placing the cardboard lid on top to keep out dust. When it has cooled add a pinch of powdered alum, stir the mixture, replace the lid, put the beaker in a cupboard where there is likely to be a fairly even temperature, and leave it for a day or two. You will then have a saturated solution above the crystals which will form.

Next pour as much as possible of the clear saturated solution into a clean beaker, taking care to leave the crystals behind. Now pour off a little solution into a small basin. Put a cardboard lid on the beaker containing the remainder, and place it in the cupboard; you will need it later. Leave the basin exposed to the air for a day or so, looking at it from time to time to see whether crystals have appeared. Small crystals will eventually form. Choose some well shaped ones and pick them out with tweezers. These are the seed crystals from which you are going to grow the big ones.

Mounting the seed crystals – This can be done by attaching the seed crystals each to a length of cotton with an adhesive such as Durofix. Place a very small quantity of the adhesive, smaller than a pin's head, on the extreme end of a piece of cotton about 6 – 9 inches long. Fix this end to the crystal and leave it lying on a piece of paper to dry. Alternatively, tie a slip knot in the end of the cotton and secure the crystal with this. You could again use a little Durofix.

Alum crystals showing octahedral (left), and cubic (right) forms and combinations of the two.

Making the solution supersaturated – Remove the cardboard from the beaker containing the saturated solution and add 16 grams of alum. (For this quantity we assume that the room temperature at which you are going to grow your crystals is about 25°C. If it is lower than this, add a little less alum; if higher, add a little more.) Put the beaker in the can of water and heat slowly, stirring the solution in the beaker until the added alum has dissolved. Then remove the beaker from the can, put the cardboard lid on again, and leave the solution to cool. The solution will then be supersaturated and ready for the seeds to be put in to grow.

'*Planting the seed*' – Make a pinhole in the centre of another cardboard lid and pass the cotton to which the seed crystal is attached through the hole from underneath. Pull it through until the crystal hangs in the middle of the solution when the lid is placed on the beaker. Fix the cotton on the upper side of the lid with a small piece of Sellotape or Plasticine but do not put the seed in the solution yet. When putting the seed in to grow, it is important not to admit particles of dust or small particles of alum powder, or this will cause unwanted crystals to appear – in addition to the crystal you hope will grow round your 'seed'. As a safeguard bring the supersaturated solution to a temperature about 3°C above the growing temperature (i.e. 3°C above room temperature). Do this by placing it in a tin of hot or cold water as necessary. By having the solution a few degrees hotter than the growing temperature, you guard to some extent against unwanted crystals growing, as small unwanted seeds will dissolve. Your chosen seed will also dissolve a little, but as it is comparatively big, this will not matter. Finally remove the cardboard now covering the beaker, replace it with the cover carrying the seed crystal, and set the beaker aside in the cupboard. Next day, the seed should have grown into quite a large crystal, a centimetre or so across. Leave it until it seems to have stopped growing. This may take several days.

Growing larger crystals – If you want to try to get your crystal to grow even bigger, repeat the experiment starting at the place headed 'Making the solution supersaturated'. Add another 16 grams of alum and proceed as before, using the crystal you have just grown as the seed. Before planting it, dip it for a second or two into warm water to dissolve any unwanted small crystals.

A slower, but perhaps easier, way to get a larger crystal is to leave the original crystal suspended in its beaker; but pierce several big holes in the lid, as this will enable the solution to evaporate and more alum will deposit slowly on the crystal. It is best to put a filter paper or a sheet of blotting paper on the top of the lid: this will exclude dust, but will not stop the water evaporating. The crystal may be left to grow in this way for several days.

When the crystal has stopped growing remove it from the solution, and wipe it quickly and carefully with damp filter paper. Do not handle it more than necessary. Store it, preferably wrapped in a piece of tissue paper, in a corked tube.

Another way to mount the seed crystal – You will need a test-tube with a small hole in the bottom. Drop the seed crystal into the test-tube so that a corner sticks out through the hole. Obviously, the seed must be larger than the hole. To support the seed in the supersaturated solution, make a hole in the cardboard the diameter of the test-tube so that the test-tube

can be passed through it and held vertically. When the crystal has grown, it will be nicely supported on the test-tube. It can be preserved by fixing a larger tube over a ring of cork round the original test-tube.

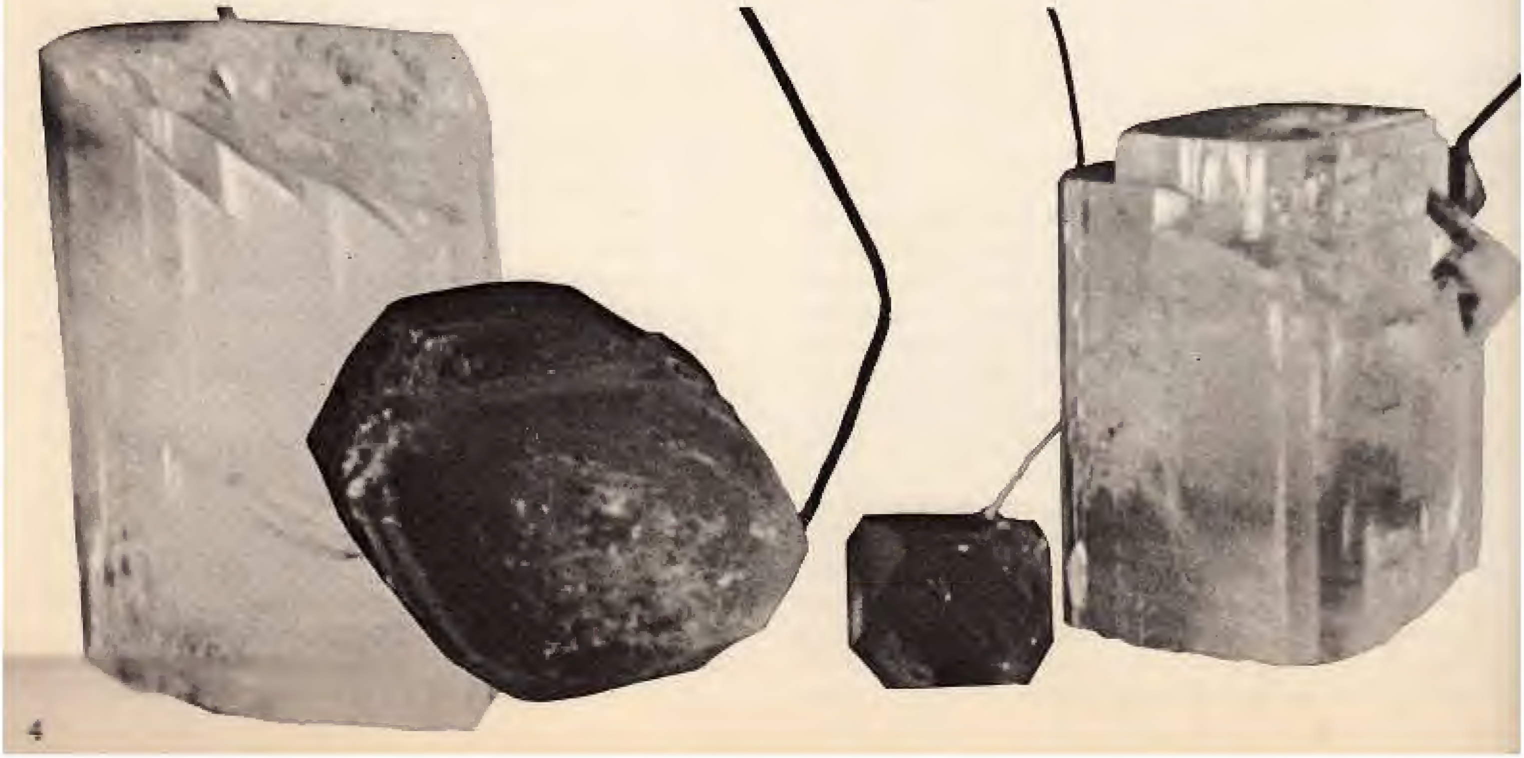
Recipes for growing other crystals – In the following recipes, the first line gives the weight of substance to warm with 100 cm^3 of water in order to make the saturated solution. The second line gives the additional weight of substance to add to this saturated solution in order to make it supersaturated. Of course, if you want to make larger volumes of solution, you must increase the weights of substance proportionately.

1 Chrome alum (purple)	60 grams per 100 cm^3 of water Add 5 grams for supersaturating.

You can make very beautiful violet-coloured crystals of a mixture of potash alum and chrome alum by mixing the growing solutions for the two alums in any proportions you wish. The greater the proportion of ordinary alum (potash alum), the paler will be the purple colour of the crystals.

2 Nickel sulphate (green)	115 grams of hexahydrate per 100 cm^3 of water Add 19 grams for supersaturating.
3 Sodium nitrate (colourless)	110 grams per 100 cm^3 of water Add 3 grams for supersaturating.

Home-grown crystals of Rochelle salt and a mixed crystal of potassium and chrome alum (second from right).



Part three

Growing crystals at home

The equipment needed to grow crystals at home: one 1 lb jam jar; a saucepan; thermometer; cotton, cardboard, and glue; a weighing pan; and, of course, the substance from which crystals are to be grown.

If you do the experiments at home, you may not have all the apparatus you would use in the laboratory, but it is often possible to find other things that will do almost as well.

1 Two 1 lb jam jars (instead of beakers).
2 A saucepan will do just as well as a tin.
3 A thermometer (0–100°C; you will have to borrow one).
4 A reel of cotton, some Durofix or other glue, and some cardboard to cover the jam jars (if they have lids these will do just as well and might even be better).
5 A gas or electric ring will be needed to heat the saucepan of water.
6 A pair of kitchen scales to weigh the crystals. Failing this, devise a balance of your own, using pennies and halfpennies as weights (1d = $\frac{1}{3}$ ounce, $\frac{1}{2}$d = $\frac{1}{4}$ ounce).
7 A measuring jug to measure the water. Failing this, use a full-sized teaspoon ($7\frac{1}{2}$ teaspoonfuls = 1 fluid ounce), a milk bottle, or a glass or cup of known volume.
8 A clean piece of cloth, to use as a filter, but make sure it is one which does not matter, for it will get badly stained.
9 A pair of tweezers to pick up the crystals (if you have none, pick them up between two knives).

Growing a large crystal of Rochelle salt (sodium potassium tartrate) from a small seed crystal. The seed crystal was suspended on a cotton thread in a saturated solution of the alum.

To prepare the saturated solution – Clean two jam jars very thoroughly. Measure out half a pint (10 fluid ounces) of water into one beaker and add 2 ounces of powdered alum. Warm the mixture to 50°C by placing the beaker in some water in the saucepan and heating the saucepan. Stir the solution well for two or three minutes. When the alum has all dissolved, pour the solution while it is still warm into the other jam jar, filtering it to remove any impurities. Put it aside to cool, placing a piece of cardboard (or a jam jar lid) on top to keep out dust. When it has cooled, add a pinch of powdered alum, stir the mixture, replace the lid, put the jam jar in a cupboard where there is likely to be a fairly even temperature, and leave it for a day or two. You will then have a saturated solution above the crystals which will form.

Next pour as much as possible of the clear saturated solution into a clean jam jar, taking care to leave the crystals behind. Now pour off a little solution into a small basin. Cover the jam jar containing the remainder and place it in the cupboard; you will need it later. Leave the basin exposed to the air for a day or so, looking at it from time to time to see whether crystals have appeared. Small crystals will eventually form. Choose some well shaped ones and pick them out with tweezers or between two knives. These are the seed crystals from which you are going to grow the big ones.

Mounting the seed crystals – This can be done by attaching the seed crystals each to a length of cotton with an adhesive such as Durofix. Place a very small quantity of the adhesive, smaller than a pin's head, on the extreme end of a piece of cotton about 6 – 9 inches long. Fix this end to the crystal and leave it lying on a piece of paper to dry. Alternatively,

tie a slip knot in the end of the cotton and secure the crystal with this. You could again use a little Durofix.

Making the solution supersaturated – Remove the cover from the jam jar containing the saturated solution and add half an ounce of alum. (For this quantity we assume that the room temperature at which you are going to grow your crystals is about 25°C. If it is lower than this, add a little less alum; if higher, add a little more.) Put the jam jar in the saucepan of water and heat slowly, stirring the solution in the jam jar until the added alum has dissolved. Then remove the jar from the saucepan, put the lid on again, and leave the solution to cool. The solution will then be supersaturated and ready for the seeds to be put in to grow.

'*Planting the seed*' – Make a pinhole in the centre of another cardboard lid or jam jar lid and pass the cotton to which the seed crystal is attached through the hole from underneath. Pull it through until the crystal hangs in the middle of the solution when the lid is placed on the jar. Fix the cotton on the upper side of the lid with a small piece of Sellotape or Plasticine, but do not put the seed in the solution yet. When putting the seed in to grow, it is important not to admit particles of dust or small particles of alum powder, or this will cause unwanted crystals to appear – in addition to the crystal you hope will grow round your 'seed'. As a safeguard bring the supersaturated solution to a temperature about 3°C above the growing temperature (i.e. 3°C above room temperature). Do this by placing it in a saucepan of hot or cold water as necessary. By having the solution a few degrees hotter than the growing temperature, you guard to some extent against unwanted crystals growing, as small unwanted seeds will dissolve. Your chosen seed will also dissolve a little, but as it is comparatively big, this will not matter. Finally remove the cover from the jam jar, replace it with the cover carrying the seed crystal, and set the jar aside in the cupboard. Next day, the seed should have grown into quite a large crystal, a centimetre or so across. Leave it until it seems to have stopped growing. This may take several days.

Growing larger crystals – If you want to try to get your crystal to grow even bigger, repeat the experiment starting at the place headed 'Making the solution supersaturated'. Add another half ounce of alum and proceed as before, using the crystal you have just grown as the seed. Before planting it, dip it for a second or two into warm water to dissolve any unwanted small crystals.

A slower, but perhaps easier, way to get a larger crystal is to leave the original crystal suspended in its jar, but pierce several big holes in the lid, as this will enable the solution to evaporate and more alum will deposit slowly on the crystal. It is best to put a filter paper or a sheet of blotting paper on the top of the lid: this will exclude dust, but will not stop the water evaporating. The crystal may be left to grow in this way for several days.

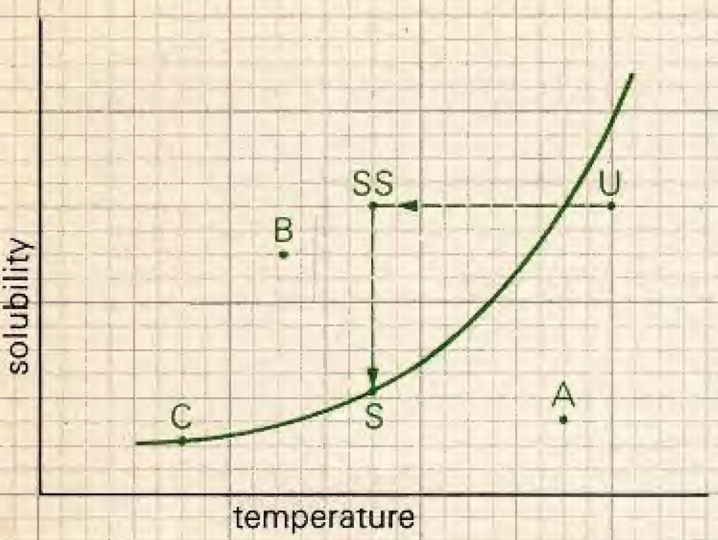
When the crystal has stopped growing remove it from the solution, and wipe it quickly and carefully with damp filter paper. Do not handle it more than necessary. Store it, preferably wrapped in a piece of tissue paper, in a corked tube.

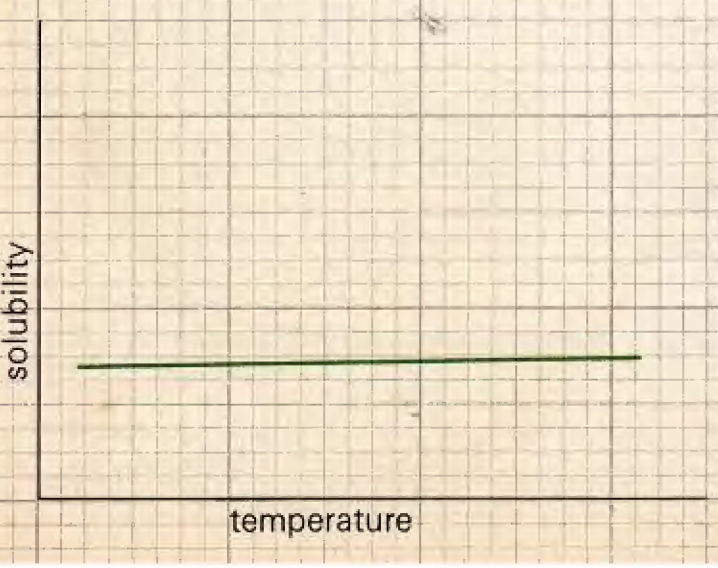
For making the other crystals (see page 4), use the following quantities:

Chrome alum	2 ounces to 3½ fluid ounces of water. (20 fluid ounces = 1 pint) Add ⅛ ounce for supersaturating.
Nickel sulphate	4 ounces to 3½ fluid ounces of water. Add a little over ½ ounce for supersaturating.
Sodium nitrate	3½ ounces to 3½ fluid ounces of water. Add ⅛ ounce for supersaturating.

Part four

How it works





How it works – The reasons why you were able to grow crystals in the way described are most easily understood by looking at the diagram. It shows the solubility in water of a substance such as alum, at various temperatures. The 'solubility' is the weight of substance required to saturate 100 grams of water at a stated temperature. At higher temperatures, the solubility is usually greater. The saturated solution you made could be represented by the point S on the diagram. When you added more substance and raised the temperature, the solution could have corresponded to the point U. When you let this cool to room temperature, it was supersaturated and could be represented by the point SS. Then you put the seed in and left it to grow. As the substance came out of the supersaturated solution and deposited on the seed crystal, the solution slowly moved back to the point S, where it was no longer supersaturated.

Questions

1 What would you call a solution represented by the point A?

2 What would happen to a seed crystal placed in a solution represented by the point A?

3 What would happen if you put a seed crystal into a solution represented by the point B? – and C?

4 (Hard question) What does the distance between S and SS represent?

Some salts are not much more soluble at high temperatures than at low. Common salt is one. Its solubility curve looks like this (left).

5 You could not grow large crystals of common salt by the method you have used for alum. Can you see why this is?

6 Can you think of a way in which large crystals of common salt might be grown?

Part five

Crystals in nature

Crystals of rock salt showing characteristic cubic shape

Quite large crystals of common salt are found in salt mines – they are cubic in shape and may be up to several inches in length. They must have been formed by the slow evaporation of saturated solutions of salt over a long period of time. The salt deposits in Cheshire are believed to be the remains of ancient seas which became landlocked and dried up about 150 million years ago. Many other substances are found as crystals in cavities in rocks. They come in a great variety of colours and shapes and any size from a pinhead to a foot in length; in rare instances even bigger. Mica has been found in crystals 12 feet in diameter, while the largest single crystal ever found – of a substance called spodumene – was 42 feet long and weighed 65 tons. Such giants are the result of very exceptional combinations of circumstances and must have grown undisturbed through many years, perhaps even centuries. If you would like to know more about chemicals in the earth and how they are deposited, take a look at the Background Book, *Chemicals and where they come from.*

If you want to see some really fine examples of natural crystals, you should visit a museum where there is a collection of minerals. Most towns have one, and you will find

Cavity in a rock partly filled with quartz crystals.

Natural crystals of diamond, most of which will be cut to make gems.

A crystal of tourmaline (a mineral containing boron) embedded in crystals of quartz. *Crown Copyright, Science Museum, London.*

many magnificent specimens in these collections. Look for crystals of clear quartz ('rock crystal'), coloured quartz (rose, smoky, amethyst, etc.), pyrites (a mineral which is a compound of iron), galena (an ore of lead), as well as crystals of precious stones such as diamond and emerald, and of semi-precious stones such as zircon and garnet. You may be fortunate enough to see specimens of the natural crystals of these, but most precious stones will have been cut by jewellers into shapes that make them sparkle in the light. These cut stones look like natural crystals, but their straight edges and flat faces have not grown naturally, as was the case with your alum crystals, but have been made and polished by the expert jeweller.

Man has often tried to grow crystals of precious stones and in recent years he has had some success. You can read the story of how diamonds were made, after many attempts spread over nearly a century, in another Background Book, *Making diamonds*. Crystals of many other minerals have now been grown in laboratories, for example crystals of ruby, sapphire, and emerald. They are called 'synthetic'. They are not 'imitations' because they consist of exactly the same

A natural quartz crystal.

A crystal of garnet — a mineral containing chromium sometimes used as a gem.
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Crystals of calcite, a mineral composed of calcium carbonate.
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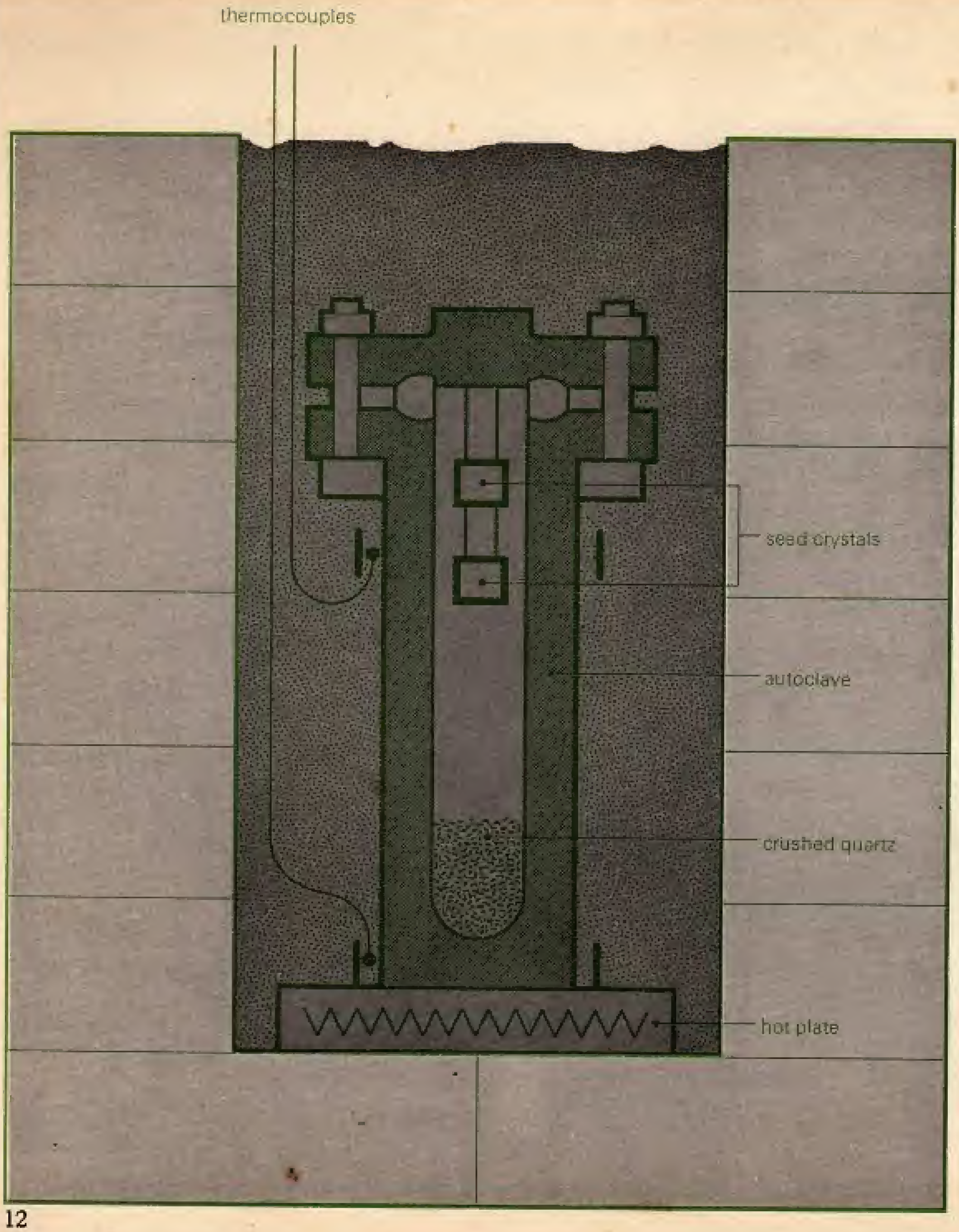

Diagram of autoclave for making large quartz crystals.

Autoclave for making quartz. The insulating material is packed in the box round the furnace as lagging to prevent heat loss.

stuff as the natural mineral, and the only difference is that they grew into crystals in a piece of laboratory apparatus instead of in the rocks of the Earth's crust. Large synthetic crystals of quartz, up to 3 – 4 inches across, are also grown in some industrial laboratories. They are used for making certain radio components. Quartz has an extremely small solubility in water and you may wonder, therefore, how it is possible to crystallize it. The secret is 'high pressure'. When it is sealed up with water in a very strong steel vessel and heated to 300 or 400 degrees centigrade, it becomes appreciably soluble and it is under these conditions that it is made to grow from seed crystals.

Some crystals can be grown by melting the substances, instead of dissolving them in water. Crystals of sapphire and ruby, and also of common salt, are grown in this way by manufacturers.

The largest quartz crystal ever made by man.
General Electric Company

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